

“DETERMINATION OF CHLORINATED ACIDS IN WATER BY GAS CHROMATOGRAPHY WITH AN ELECTRON CAPTURE DETECTOR”
By EPA 515.1 Revision 4.1 1995

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Facility Name: _____ VELAP ID _____

Assessor Name: _____ Analyst Name: _____ Inspection Date _____

Relevant Aspect of Standards**Method
Reference****Y****N****N/A****Comments**

Records Examined: SOP Number/ Revision/ Date _____ Analyst: _____

Sample ID: _____ Date of Sample Preparation: _____ Date of Analysis: _____

Were sample containers cleaned by rinsing with the last solvent used, washing with detergent and hot water, rinsing with dilute acid, rinsing with tap water, rinsing with reagent water, and finally heated in at 400°C for 1 hour?

4.1.1
6.1

Was the glass wool acid washed and then heated at 450°C for four hours prior to use?

6.9

Was the Sodium Sulfate (Na₂SO₄) first heated at 450°C for four hours?

7.3

Was the Sodium Sulfate (Na₂SO₄) then acidified by slurring with ethyl ether followed by the addition of sulfuric acid?

7.3

Was the ethyl ether then vacuumed off the Sodium Sulfate (Na₂SO₄), and a portion of the acidified Sodium Sulfate (Na₂SO₄) then slurried with reagent water and confirmed to have a pH less than 4 prior to use?

7.3

Was the Sodium Sulfate (Na₂SO₄) stored at 130°C?

7.3

Was the Florisil activated by heating at 150°C for between 24 and 48 hours prior to use?

7.18

If residual chlorine was present, was sodium thiosulfate added to sample bottles prior to collection?

8.2.1

Were samples iced or refrigerated at 4°C and stored away from lights between collection and extraction?

8.2.3

Were sample extracts stored at 4°C away from light?

8.3.1

Were surrogate compound recoveries determined to be 70-130% for each sample and blank?

9.1
9.5.1

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Were internal standard (IS) peak areas OR heights monitored for each sample and blank?	9.1				
Did the IS responses of all samples not deviate from the calibration check IS responses by more than $\pm 30\%$?	9.6.1				
Were MDLs determined for each analyte?	9.1				
Did the analyses of LRBs/MBs demonstrate that all glassware and reagent interferences are "under control"?	9.2				
Was an LFB analyzed with each sample extraction and with at least every 20 samples?	9.7.1				
After the accumulation of 20-30 LFB results, were LFBs assessed against criteria of ± 3 Standard Deviations?	9.7.2				
Were calibration checks assessed against criteria of $\pm 20\%$?	10.2.4				
Were sample matrix spikes analyzed at a minimum frequency of 10% of samples or at least one per sample set?	9.8.1				
Were the matrix spikes assessed against criteria of 65-135%?	9.8.2				
Were calibration standards prepared at a minimum of three concentrations?	10.2.1				
Were Average Response Factors (RFs) used for quantitation only if the RF value over the working range was 20% RSD or less?	10.23				
Manual Hydrolysis, Preparation, and Extraction					
Did LRB/MBs and LFBs have the same preservatives (eg sodium thiosulfate) added as the associated samples?	11.1.1				
Did samples have NaCl added to them followed by shaking?	11.1.2				
Did samples have 6 N NaOH added to them followed by shaking?	11.1.3				
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Were the samples, after the addition of NaOH, determined to have pHs≥12?	11.1.3				
Were samples then added to separatory funnels?	11.1.3				
Were samples allowed to sit at room temperature for one hour?	11.1.3				
Were samples containers rinsed with methylene chloride into the corresponding separatory funnels?	11.1.4				
Were separatory funnels shaken for two minutes?	11.1.4				
Were water phases and organic phases allowed to separate for a minimum of 10 minutes?	11.1.4				
Were methylene chloride layers discarded?	11.1.4				
Were the above 4 steps repeated twice more?	11.1.5				
Did the water samples have 12 N H ₂ SO ₄ added followed by shaking?	11.1.6				
Were samples then determined to have a pH<2?	11.1.6				
Was ethyl ether added to samples followed by two minutes of vigorous shaking?	11.1.7				
Were organic layers and water layers allowed to separate for a minimum of 10 minutes?	11.1.7				
Were extracts dried with anhydrous Sodium Sulfate Na ₂ SO ₄ for approximately two hours?	11.1.7				
Were the initial sample volumes determined with the aqueous phases?	11.1.8				
Automated Hydrolysis, Preparation, and Extraction					
Did LRB/MBs and LFBs have the same preservatives (eg sodium thiosulfate) added as the associated samples?	11.2.1				
Did samples have NaCl added to them followed by shaking?	11.2.2				
Did samples have 6 N NaOH added to them followed by shaking on a mechanical mixer/shaker for 1 hour?	11.2.3				
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5 Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Was methylene chloride added to samples followed by shaking until no pressure evolves?	11.2.4				
Were samples next shaken or tumbled for one hour?	11.2.4				
Were the organic phases then discarded?	11.2.5				
Did the water samples have 12 N H ₂ SO ₄ added followed by shaking?	11.2.6				
Were samples then determined to have a pH<2?	11.2.6				
Was ethyl ether added to samples followed by shaking until no pressure evolves?	11.2.7				
Were samples then shaken or tumbled for one hour?	11.2.7				
Were aqueous phases and organic phases allowed to separate for a minimum of 10 minutes?	11.2.8				
Were extracts dried with anhydrous Sodium Sulfate Na ₂ SO ₄ for approximately two hours?	11.2.8				
Were the initial sample volumes determined with the aqueous phases?	11.2.9				
Extraction Concentration					
Were dried extracts filter through glass wool into a Kuderna-Danish apparatus?	11.3.2				
Were extracts concentrated on K-D apparatus at 60-65°C until 1 mL was reached?	11.3.3				
Was MTBE added to the K-D apparatus?	11.3.4				
Were extracts concentrated to approximately 0.50 mL?	11.3.4				
Was methanol added to the extracts?	11.3.4				
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Esterification of Acids Using Gaseous Diazomethane					
Was diazomethane bubble through extract for one minute?	11.4.2				
Did samples turn yellow and remain yellow for at least two minutes?	11.4.2				
Were extracts stored at room temperature for 30 minutes?	11.4.3				
Esterification of Acids Using Diazomethane Solution--Alternative					
Was a Diazald and 37% KOH solution purged by nitrogen into the collection vial for 30 minutes?	11.5.2				
Was liquid diazomethane solution added to the extracts?	11.5.3				
Did samples turn yellow and remain yellow for at least two minutes?	11.5.3				
Esterification of Acids using TMSD--Alternative					
Was the procedure carried after the procedure in 11.4?	11.6.1				
Was a 2 M TMSD solution added to each sample extract?	11.6.2				
Were the extracts heated for one hour at 50°C?	11.6.3				
Analytical Measurement					
If internal standard calibration was used, was internal standard added after extraction?	11.8.3				
Notes/Comments:					